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April 25, 2016

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APPLICATION NUMBER: 14/926,377

FILING DATE: October 29, 2015

THE COUNTRY CODE AND NUMBER OF YOUR PRIORITY APPLICATION, TO BE USED FOR FILING ABROAD UNDER THE PARIS CONVENTION, IS US14/926,377

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Under Secretary of Commerce for Intellectual Property
and Director of the United States Patent and Trademark Office**



JOHN A BURSON

Certifying Officer

Electronic Acknowledgement Receipt

EFS ID:	23929317
Application Number:	14926377
International Application Number:	
Confirmation Number:	5582
Title of Invention:	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME
First Named Inventor/Applicant Name:	James Timothy BRISTOW
Customer Number:	35161
Filer:	Bruce D. Gray./Silvia Gomez
Filer Authorized By:	Bruce D. Gray.
Attorney Docket Number:	727440-01028
Receipt Date:	29-OCT-2015
Filing Date:	
Time Stamp:	13:43:11
Application Type:	Utility under 35 USC 111(a)

Payment information:

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File Listing:

Document Number	Document Description	File Name	File Size(Bytes)/ Message Digest	Multi Part /.zip	Pages (if appl.)
1	Transmittal of New Application	aia0015TRANSMITTAL.pdf	275556 816f5549b19308867126e189461e309df13e5fb8	no	2

Warnings:

Information:

2	Fee Worksheet (SB06)	sb0017.pdf	256515 01db9aa7e55f329cbb6eaa6a0edc0eff834ec8ef	no	2
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Warnings:

Information:

3	Application Data Sheet	aia0014ADS.pdf	1561162 817e7c92fcc6320755f2164dd1a8b5348f3165b	no	7
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Warnings:

Information:

4		APPLICATION.pdf	985493 750bb79513b89ff7a50daec182a42495387a2dc	yes	27
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Multipart Description/PDF files in .zip description

Document Description	Start	End
Specification	1	18
Claims	19	22
Abstract	23	23
Drawings-only black and white line drawings	24	27

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Information:

5	Fee Worksheet (SB06)	fee-info.pdf	37304 f4b42b9479994d0381e5b4085fc123bff76ed705	no	2
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Total Files Size (in bytes):

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If a new application is being filed and the application includes the necessary components for a filing date (see 37 CFR 1.53(b)-(d) and MPEP 506), a Filing Receipt (37 CFR 1.54) will be issued in due course and the date shown on this Acknowledgement Receipt will establish the filing date of the application.

National Stage of an International Application under 35 U.S.C. 371

If a timely submission to enter the national stage of an international application is compliant with the conditions of 35 U.S.C. 371 and other applicable requirements a Form PCT/DO/EO/903 indicating acceptance of the application as a national stage submission under 35 U.S.C. 371 will be issued in addition to the Filing Receipt, in due course.

New International Application Filed with the USPTO as a Receiving Office

If a new international application is being filed and the international application includes the necessary components for an international filing date (see PCT Article 11 and MPEP 1810), a Notification of the International Application Number and of the International Filing Date (Form PCT/RO/105) will be issued in due course, subject to prescriptions concerning national security, and the date shown on this Acknowledgement Receipt will establish the international filing date of the application.

PTO/AIA/15 (03-13)

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UTILITY PATENT APPLICATION TRANSMITTAL (Only for new nonprovisional applications under 37 CFR 1.53(b))	Attorney Docket No.	727440-01028
	First Named Inventor	James Timothy BRISTOW
	Title	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROSECO FOR ITS PREPARATION AND
	Express Mail Label No.	

APPLICATION ELEMENTS <i>See MPEP chapter 600 concerning utility patent application contents.</i>	Commissioner for Patents P.O. Box 1450 Alexandria, VA 22313-1450
1. ☒ Fee Transmittal Form (PTO/SB/17 or equivalent)	ACCOMPANYING APPLICATION PAPERS
2. ☐ Applicant asserts small entity status. See 37 CFR 1.27	10. ☐ Assignment Papers (cover sheet & document(s)) Name of Assignee _____
3. ☐ Applicant certifies micro entity status. See 37 CFR 1.29. Applicant must attach form PTO/SB/15A or B or equivalent.	11. ☐ 37 CFR 3.73(c) Statement ☐ Power of Attorney (when there is an assignee)
4. ☒ Specification [Total Pages <u>24</u>] Both the claims and abstract must start on a new page. (See MPEP § 608.01(a) for information on the preferred arrangement)	12. ☐ English Translation Document (if applicable)
5. ☒ Drawing(s) (35 U.S.C. 113) [Total Sheets <u>4</u>]	13. ☐ Information Disclosure Statement (PTO/SB/08 or PTO-1449) ☐ Copies of citations attached
6. Inventor's Oath or Declaration [Total Pages _____] (including substitute statements under 37 CFR 1.64 and assignments serving as an oath or declaration under 37 CFR 1.63(e))	14. ☐ Preliminary Amendment
a. ☐ Newly executed (original or copy)	15. ☐ Return Receipt Postcard (MPEP § 503) (Should be specifically itemized)
b. ☐ A copy from a prior application (37 CFR 1.63(d))	16. ☐ Certified Copy of Priority Document(s) (if foreign priority is claimed)
7. ☒ Application Data Sheet * See note below. See 37 CFR 1.76 (PTO/AIA/14 or equivalent)	17. ☐ Nonpublication Request Under 35 U.S.C. 122(b)(2)(B)(i). Applicant must attach form PTO/SB/35 or equivalent.
8. CD-ROM or CD-R in duplicate, large table, or Computer Program (Appendix) ☐ Landscape Table on CD	18. ☐ Other: _____ _____ _____ _____
9. Nucleotide and/or Amino Acid Sequence Submission (if applicable, items a. – c. are required)	
a. ☐ Computer Readable Form (CRF)	
b. ☐ Specification Sequence Listing on: i. ☐ CD-ROM or CD-R (2 copies); or ii. ☐ Paper	
c. ☐ Statements verifying identity of above copies	

***Note:** (1) Benefit claims under 37 CFR 1.78 and foreign priority claims under 1.55 **must** be included in an Application Data Sheet (ADS).
(2) For applications filed under 35 U.S.C. 111, the application must contain an ADS specifying the applicant if the applicant is an assignee, person to whom the inventor is under an obligation to assign, or person who otherwise shows sufficient proprietary interest in the matter. See 37 CFR 1.46(b).

19. CORRESPONDENCE ADDRESS					
☒ The address associated with Customer Number: <u>35161</u> OR ☐ Correspondence address below					
Name					
Address					
City	State	Zip Code			
Country	Telephone	Email			
Signature	/Bruce D. Gray/		Date	29 October 2015	
Name (Print/Type)	Bruce D. Gray		Registration No. (Attorney/Agent)	35,799	

This collection of information is required by 37 CFR 1.53(b). The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.11 and 1.14. This collection is estimated to take 12 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (*i.e.*, GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
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PTO/SB/17 (03-13)

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FEE TRANSMITTAL

Complete if known

- ☐ Applicant asserts small entity status. See 37 CFR 1.27.
- ☐ Applicant certifies micro entity status. See 37 CFR 1.29.
Form PTO/SB/15A or B or equivalent must either be enclosed or have been submitted previously.

Application Number Unassigned
Filing Date 29 October 2015
First Named Inventor James Timothy BRISTOW
Examiner Name Unassigned
Art Unit Unassigned
Practitioner Docket No. 727440-01028

TOTAL AMOUNT OF PAYMENT (\$)

METHOD OF PAYMENT (check all that apply)

- ☐ Check ☒ Credit Card ☐ Money Order ☐ None ☐ Other (please identify): _____
- ☒ Deposit Account Deposit Account Number: 04-1061 Deposit Account Name: _____
For the above-identified deposit account, the Director is hereby authorized to (check all that apply):
☒ Charge fee(s) indicated below ☐ Charge fee(s) indicated below, except for the filing fee
☐ Charge any additional fee(s) or underpayment of fee(s) under 37 CFR 1.16 and 1.17 ☒ Credit any overpayment of fee(s)

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FEE CALCULATION

1. BASIC FILING, SEARCH, AND EXAMINATION FEES (U = undiscounted fee; S = small entity fee; M = micro entity fee)

Application Type	FILING FEES			SEARCH FEES			EXAMINATION FEES			Fees Paid (\$)
	U (\$)	S (\$)	M (\$)	U (\$)	S (\$)	M (\$)	U (\$)	S (\$)	M (\$)	
Utility	280	140*	70	600	300	150	720	360	180	1600.00
Design	180	90	45	120	60	30	460	230	115	
Plant	180	90	45	380	190	95	580	290	145	
Reissue	280	140	70	600	300	150	2,160	1,080	540	
Provisional	260	130	65	0	0	0	0	0	0	

* The \$140 small entity status filing fee for a utility application is further reduced to \$70 for a small entity status applicant who files the application via EFS-Web.

2. EXCESS CLAIM FEES

Fee Description	Undiscounted Fee (\$)	Small Entity Fee (\$)	Micro Entity Fee (\$)
Each claim over 20 (including Reissues)	80	40	20
Each independent claim over 3 (including Reissues)	420	210	105
Multiple dependent claims	780	390	195
Total Claims			
-20 or HP = _____ x _____ = _____	Fee Paid (\$)		
HP = highest number of total claims paid for, if greater than 20.		Multiple Dependent Claims	
Indep. Claims		Fee (\$)	Fee Paid (\$)
-3 or HP = _____ x _____ = _____	Fee Paid (\$)		
HP = highest number of independent claims paid for, if greater than 3.			

3. APPLICATION SIZE FEE

If the specification and drawings exceed 100 sheets of paper (excluding electronically filed sequence or computer listings under 37 CFR 1.52(e)), the application size fee due is \$400 (\$200 for small entity) (\$100 for micro entity) for each additional 50 sheets or fraction thereof. See 35 U.S.C. 41(a)(1)(G) and 37 CFR 1.16(s).

Total Sheets	Extra Sheets	Number of each additional 50 or fraction thereof	Fee (\$)	Fee Paid (\$)
_____ - 100 = _____ / 50 = _____ (round up to a whole number)		_____ x _____ = _____		

4. OTHER FEE(S)

Non-English specification, \$130 fee (no small or micro entity discount) _____ **Fees Paid (\$)**

Non-electronic filing fee under 37 CFR 1.16(t) for a utility application, \$400 fee (\$200 small or micro entity) _____

Other (e.g., late filing surcharge): _____

SUBMITTED BY

Signature	/Bruce D. Gray/	Registration No. (Attorney/Agent)	35,799	Telephone	2026596973
Name (Print/Type)	Bruce D. Gray			Date	29 October 2015

This collection of information is required by 37 CFR 1.136. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 30 minutes to complete, including gathering, preparing, and submitting the completed application form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.

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2. A record from this system of records may be disclosed, as a routine use, in the course of presenting evidence to a court, magistrate, or administrative tribunal, including disclosures to opposing counsel in the course of settlement negotiations.
3. A record in this system of records may be disclosed, as a routine use, to a Member of Congress submitting a request involving an individual, to whom the record pertains, when the individual has requested assistance from the Member with respect to the subject matter of the record.
4. A record in this system of records may be disclosed, as a routine use, to a contractor of the Agency having need for the information in order to perform a contract. Recipients of information shall be required to comply with the requirements of the Privacy Act of 1974, as amended, pursuant to 5 U.S.C. 552a(m).
5. A record related to an International Application filed under the Patent Cooperation Treaty in this system of records may be disclosed, as a routine use, to the International Bureau of the World Intellectual Property Organization, pursuant to the Patent Cooperation Treaty.
6. A record in this system of records may be disclosed, as a routine use, to another federal agency for purposes of National Security review (35 U.S.C. 181) and for review pursuant to the Atomic Energy Act (42 U.S.C. 218(c)).
7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (*i.e.*, GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
8. A record from this system of records may be disclosed, as a routine use, to the public after either publication of the application pursuant to 35 U.S.C. 122(b) or issuance of a patent pursuant to 35 U.S.C. 151. Further, a record may be disclosed, subject to the limitations of 37 CFR 1.14, as a routine use, to the public if the record was filed in an application which became abandoned or in which the proceedings were terminated and which application is referenced by either a published application, an application open to public inspection or an issued patent.
9. A record from this system of records may be disclosed, as a routine use, to a Federal, State, or local law enforcement agency, if the USPTO becomes aware of a violation or potential violation of law or regulation.

PA-0413

A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME

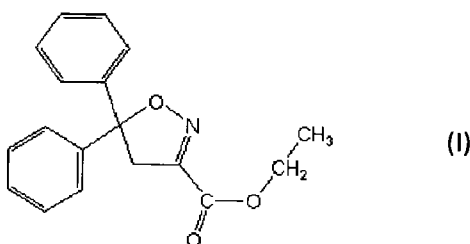
BACKGROUND

Field

5 The present disclosure relates to a crystalline form of ethyl 4,5-dihydro-5,5-diphenyl -3-isoxazolecarboxylate (isoxadifen-ethyl), to its preparation processes and to its use in agrochemical preparations.

Description of Related Art

10 Ethyl 4,5-dihydro-5,5-diphenyl-3-isoxazolecarboxylate (isoxadifen-ethyl) is a potent safener for sulfonylurea herbicides. Isoxadifen-ethyl has molecular formula of $C_{18}H_{17}NO_3$. Its chemical structure is:



15 It is typically used to reduce the phototoxicity of sulfonylurea herbicides when applied on crops, such as maize, wheat and cereals.

20 The commercially available isoxadifen-ethyl, which is usually manufactured by the process described in U.S. Patent No. 5,516,750, which is incorporated herein by reference for all purposes, is present in an amorphous state, the melting point which is about 78°C -81°C.

SUMMARY

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It has been found that isoxadifen-ethyl in amorphous state is highly unstable. It will generally undergo significant hydrolysis when dissolved or dispersed in water. Furthermore, hydrolysis can occur during storage, particularly where the compound is exposed to moisture. As a result, the stability of isoxadifen-ethyl is of great concern with respect to providing the commercially available formulations. Therefore, there is a need to provide a novel form of isoxadifen-ethyl with increased stability in formulations.

Accordingly, in an embodiment, the invention provides a novel crystalline form of isoxadifen-ethyl, termed "crystalline modification I", and a process for its preparation, as well as agrochemical compositions containing it, and methods for using it in agrochemical applications, such as methods for applying it to plants, plant loci, and plant parts. The novel crystalline modification I has been found to have increased stability relative to the amorphous form. Accordingly, in an embodiment, the invention also provides compositions for controlling undesirable plant growth, such as weeds, comprising the crystalline modification I of isoxadifen-ethyl with sulfonylurea herbicides, as a mixture with auxiliaries and carriers. The use of the crystalline modification I of isoxadifen-ethyl with sulfonylurea herbicides in the control of undesired plant growth and a method for the same are also provided by embodiments of the invention.

BRIEF DESCRIPTION OF DRAWINGS

Various features and aspects of the embodiments of the invention disclosed herein can be more clearly understood by reference to the drawings, which are intended to exemplify and illustrate, but not to limit, the scope of the invention, and wherein:

FIG. 1 is an infrared (IR) spectra of an embodiment of crystalline modification I of isoxadifen-ethyl;

FIG. 2 is a powder X-ray diffractogram of an embodiment of crystalline modification I of isoxadifen-ethyl;

FIG. 3 is a graph showing the results of a Differential Scanning Calorimetry (DSC) scan of an embodiment of crystalline modification I of isoxadifen-ethyl; and

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FIG. 4 is a graph showing the results of a powder X-ray diffractogram of amorphous isoxadifen-ethyl.

DETAILED DESCRIPTION OF SPECIFIC EMBODIMENTS

The invention can be more clearly understood by reference to the following detailed description of specific embodiments thereof, which is intended to illustrate, but not limit, the scope of the appended claims.

It has been found that an embodiment of the crystalline modification I of isoxadifen-ethyl has a significant increase in its stability when mixing with sulfonylurea herbicides in formulations that are otherwise similar to current commercially available formulations, which contain the amorphous form of isoxadifen-ethyl. In addition, it has been found that the crystalline modification I of isoxadifen-ethyl is easier to comminute or grain compared to amorphous isoxadifen-ethyl prepared in accordance with the disclosure of U.S. Patent No. 5,516,750. This allows the preparation of commercial formulations such as suspension concentrates (SC), oil-based suspension concentrates (OD), water-dispersible granules (WG) and water-soluble granules (SG). Hence, it is possible to prepare any formulations of isoxadifen-ethyl in crystalline modification I when mixing with sulfonylurea herbicides, as will be disclosed hereinafter.

By virtue of its high stability, the crystalline modification I of isoxadifen-ethyl when mixing with sulfonylurea herbicides is highly suitable for preparing compositions for controlling undesirable weeds.

According to a first aspect of the invention, a crystalline modification I of isoxadifen-ethyl is provided, exhibiting at least 3 of the following reflexes as 2θ values, in any combination thereof, in an X-ray powder diffractogram recorded using Cu—K α radiation at 25 °C:

$$2\theta = 11.027 \pm 0.2 \quad (1)$$

$$2\theta = 17.841 \pm 0.2 \quad (2)$$

$$2\theta = 19.492 \pm 0.2 \quad (3)$$

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$$2\theta = 19.841 \pm 0.2 \quad (4)$$

$$2\theta = 20.583 \pm 0.2 \quad (5)$$

$$2\theta = 22.151 \pm 0.2 \quad (6)$$

$$2\theta = 22.920 \pm 0.2 \quad (7)$$

$$5 \quad 2\theta = 23.541 \pm 0.2 \quad (8)$$

$$2\theta = 24.801 \pm 0.2 \quad (9)$$

$$2\theta = 25.775 \pm 0.2 \quad (10)$$

$$2\theta = 26.895 \pm 0.2 \quad (11)$$

$$2\theta = 27.813 \pm 0.2 \quad (12)$$

$$10 \quad 2\theta = 29.408 \pm 0.2 \quad (13)$$

$$2\theta = 30.065 \pm 0.2 \quad (14)$$

$$2\theta = 31.228 \pm 0.2 \quad (15)$$

$$2\theta = 33.502 \pm 0.2 \quad (16)$$

15 The crystalline modification I of isoxadifen-ethyl of an embodiment of the invention is characterized by an X-ray powder diffractogram having at least three of the reflexes indicated above, in any combination. This means that the crystalline modification I of an embodiment of isoxadifen-ethyl can contain 3 or more of the reflexes indicated above in any combination of reflexes. For example, the crystalline modification I of an
20 embodiment of isoxadifen-ethyl can contain at least reflexes (1), (2), and (3), or at least reflexes (2), (3), and (4), or reflexes (1), (3), and (4), or any other combination of at least three reflexes. Preferably, the crystalline modification I is one having at least four of the
aforementioned reflexes, more preferably at least five, six or seven, or eight of said
reflexes, again in any combination. An X-ray powder diffractogram of the crystalline
25 modification I of isoxadifen-ethyl is shown in Figure 2, which will be described in detail hereinafter.

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According to a preferred embodiment the crystalline form A exhibits at least 3, 4, or 5 or all of the reflexes, in any combination, from the following:

	$2\theta = 11.027 \pm 0.2$	(1)
5	$2\theta = 17.841 \pm 0.2$	(2)
	$2\theta = 19.492 \pm 0.2$	(3)
	$2\theta = 19.841 \pm 0.2$	(4)
	$2\theta = 24.801 \pm 0.2$	(9)
	$2\theta = 25.775 \pm 0.2$	(10)
10	$2\theta = 26.895 \pm 0.2$	(11)
	$2\theta = 27.813 \pm 0.2$	(12)

The crystalline modification I of isoxadifen-ethyl according to an embodiment of the invention may be further characterized by Infrared (IR) spectroscopy. The IR spectrum of the crystalline modification I is showed in Figure 1 with characteristic bands at one or more of 2984.31, 2928.19, 2160.89, 1978.81 and 1716.65 cm^{-1} .

All IR spectra were obtained using the following acquisition parameters:

FT-IR spectrometer	Bruker Tensor37
Diamond ATR unit	from Specac
Wavelength range	550 - 4000 cm^{-1}
Resolution	4 cm^{-1}
Number of scans	16

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The crystalline modification I of isoxadifen-ethyl according to an embodiment of the invention may be further characterized by differential scanning calorimetry (DSC) (Figure 3). The DSC spectrum of the crystalline modification I of isoxadifen-ethyl exhibits an endothermic peak at 86.5 °C as shown in Figure 3.

5

Methods for preparing amorphous isoxadifen-ethyl are known in the art. Amorphous isoxadifen-ethyl is manufactured and available on a commercial scale. A particularly suitable method for preparing amorphous isoxadifen-ethyl is described in U.S. Patent No. 5,516,750.

10

According to an embodiment of the invention, the crystalline modification I of isoxadifen-ethyl can be obtained by the processes below:

Isoxadifen-ethyl in amorphous state is dissolved and then crystallized from solvents.

15 In one aspect, the present invention provides a process for preparing a crystalline modification I of isoxadifen-ethyl comprising steps of:

i) preparing a solution of an amorphous isoxadifen-ethyl in a solvent;

ii) effecting crystallization of isoxadifen-ethyl from the solution to obtain a solid precipitate; and

20 iii) isolating the solid precipitate.

Suitable solvents for preparing isoxadifen-ethyl crystalline modification I include acetonitrile, halogenated hydrocarbons (for example, chlorobenzene, bromobenzene, dichlorobenzene, chlorotoluene and trichlorobenzene), ethers (for example, ethyl propyl ether, n-butyl ether, anisole, phenetole, cyclohexyl methyl ether, dimethyl ether, diethyl ether, dimethyl glycol, diphenyl ether, dipropyl ether, diisopropyl ether, di-n-butyl ether, diisobutyl ether, diisoamyl ether, ethylene glycol dimethyl ether, isopropyl ethyl ether, methyl tert-butyl ether, tetrahydrofuran, methyltetrahydrofuran, dioxane, dichlorodiethyl

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ether, methyl-tetrahydrofuran, polyethers of ethylene oxide and/or propylene oxide), nitrated hydrocarbons (for example, nitromethane, nitroethane, nitropropane, nitrobenzene, chloronitrobenzene and o-nitrotoluene), aliphatic, cycloaliphatic or aromatic hydrocarbons (for example, pentane, n-hexane, n-heptane, n-octane, nonane), cymene, petroleum
5 fractions having a boiling range of from 70 °C to 190 °C, cyclohexane, methylcyclohexane, petroleum ether, ligroin, octane, benzene, toluene and xylene), esters (for example, malonates, acetic acid n-butyl ester (n-butyl acetate), methyl acetate, ethyl acetate, isobutyl acetate, dimethyl carbonate, diethyl carbonate, dibutyl carbonate and ethylene carbonate), and aliphatic alcohols (for example, methanol, ethanol, n-propanol, isopropanol, n-butanol
10 and tert-amyl alcohol), and mixtures of any of these.

Preferred solvents include acetonitrile, ethers, aromatic hydrocarbons (such as benzene, toluene, xylene, chlorobenzene), esters and aliphatic alcohols and mixtures thereof. Particularly preferred solvents or solvent mixtures include isopropanol, toluene, methyl-tetrahydrofuran, diethyl carbonate, chlorobenzene, n-butyl acetate, isobutyl acetate,
15 n-butanol, methanol, ethanol, ethyl malonate, methyl t-butyl ether, and mixtures of these, particularly mixtures of toluene and butanol, mixtures of toluene and n-butyl acetate, mixtures of ethyl malonate and methyl t-butyl ether, as well as mixtures of butyl acetate and methyl t-butyl ether. Solvent mixtures of more than 2 or 3 or 4 components are also envisaged by embodiments of the invention.

20 In the invention, it is preferred that the solvent comprises an alcohol and/or acetonitrile, including mixtures thereof, with other solvents or particularly with each other.

Hence, according to a preferred embodiment in step (i), amorphous isoxadifen-ethyl is dissolved in a solvent comprising methanol, or acetonitrile, or a mixture thereof.

25 In step (ii), isoxadifen-ethyl is crystallized from the solution. Techniques for effecting crystallization of isoxadifen-ethyl from the solution are known to those skilled in the art. For example, in an embodiment where the solution in step (i) is formed at elevated temperatures, crystallization may be effected by cooling the solution to room or ambient temperature. In one preferred embodiment, crystallization is effected by concentrating the

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solution formed in step (i) of the process by removal of solvent. Alternatively, or in addition thereto, seed crystals, in particular seed crystals of the aforementioned crystalline modification I of isoxadifen-ethyl, may be added to the solution formed in step (i), to facilitate and/or enhance crystallization.

5 It is preferred that the solid precipitate of isoxadifen-ethyl recovered during the crystallization stage is washed with a solvent for one or more times. Preferably, the solvent employed in the washing stage consists of one or more components of the solvent employed for forming the solution in step (i), as described hereinbefore. Methanol and acetonitrile, and mixtures thereof are particularly suitable solvents for washing the
10 recovered solid of isoxadifen-ethyl.

 The invention, in an embodiment, also relates to a composition comprising the crystalline modification I of isoxadifen-ethyl and a herbicidally effective amount of sulfonylurea herbicides. The weight ratio of the isoxadifen-ethyl to sulfonylurea herbicides varies within wide limits and is preferably in a range of about 1:10 to about 10:1, in
15 particular in a range of about 1:10 to about 5:1.

 The amount of sulfonylurea herbicides in the formulation can be less than about 70% by weight of the formulation, preferably less than about 50% by weight of the formulation, particularly preferably about 25% by weight of the formulation.

 The use of amorphous isoxadifen-ethyl as a safener for sulfonylurea herbicides is
20 known in the art and is used on a commercial scale. The crystalline modification I of isoxadifen-ethyl is also active in reducing the phototoxicity of sulfonylurea herbicides when controlling weeds. Techniques of formulating and applying amorphous isoxadifen-ethyl and sulfonylurea herbicides are known in the art. Isoxadifen-ethyl in the crystalline modification I of the present invention may be formulated and applied in an
25 analogous manner with sulfonylurea herbicides.

 Accordingly, in a further aspect, the invention provides a herbicidal composition comprising isoxadifen-ethyl in the crystalline modification I, as defined hereinbefore, with sulfonylurea herbicides.

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Accordingly, an embodiment of the invention furthermore provides processes for preparing compositions for controlling weeds using the crystalline modification I of isoxadifen-ethyl together with sulfonylurea herbicides.

5

The crystalline modification I of isoxadifen-ethyl together with sulfonylurea herbicides can be incorporated in a known manner to the customary formulations, such as suspension concentrates (SC), oil-based suspension concentrates (OD), water-soluble granules (SG), dispersible concentrates (DC), emulsifiable concentrates (EC), emulsion
10 seed dressings, suspension seed dressings, granules (GR), microgranules (MG), suspoemulsions (SE) and water-dispersible granules (WG) using herbicidally acceptable auxiliaries, carriers and solvents.

These formulations are prepared by mixing the crystalline modification I of isoxadifen-ethyl and sulfonylurea herbicides in amounts of each analogous to those used
15 for amorphous isoxadifen-ethyl with customary additives, for example, liquid diluents, solid diluents, wetting agents, dispersants, thickening agents, antifoaming agent and other formulation ingredients.

Sulfonylurea herbicides suitable for safening by the crystalline modification I of isoxadifen-ethyl include, but are not limited to, amidosulfuron, azimsulfuron, bensulfuron-methyl, chlorimuron-ethyl, chlorsulfuron, cinosulfuron, cyclosulfamuron, ethametsulfuron-methyl, ethoxysulfuron, flupyrsulfuron-methyl, flazasulfuron, foramsulfuron, halosulfuron-methyl, imazosulfuron, iodosulfuron-methyl, mesosulfuron-methyl, metsulfuron-methyl, nicosulfuron, oxasulfuron, primisulfuron-methyl, prosulfuron, pyrazosulfuron-ethyl, rimsulfuron,
20 sulfometuron-methyl, sulfosulfuron, thifensulfuron-methyl, triasulfuron, tribenuron-methyl, trifloxysulfuron, triflusulfuron-methyl, tritosulfuron and salts thereof, and mixtures thereof.
25

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The following sulfonylureas are preferred for safening by the crystalline modification I of isoxadifen-ethyl in accordance with an embodiment of the invention: bensulfuron-methyl, flupyrsulfuron-methyl, metsulfuron-methyl, nicosulfuron, rimsulfuron, sulfometuron-methyl, thifensulfuron-methyl, tribenuron-methyl, halosulfuron-methyl, 5 iodosulfuron-methyl, mesosulfuron-methyl and salts thereof, and mixtures thereof.

Liquid diluents include, but are not limited to, water, N,N-dimethylamide, dimethyl sulfoxide, N-alkylpyrrolidone, ethylene glycol, polypropylene glycol, propylene carbonate, dibasic esters, paraffines, alkylbenzenes, alkyl naphthalenes, glycerine, triacetine, oils of olive, castor, linseed, sesame, corn, peanut, cotton-seed, soybean, 10 rape-seed and coconut, ketones such as cyclohexanone, 2-heptanone, isophorone and 4-hydroxy-4-methyl-2-pentanone, acetates such as hexyl acetate, heptyl acetate and octyl acetate, and alcohols such methanol, cyclohexanol, decanol, benzyl and tetrahydrofurfuryl alcohol, and mixtures of these.

Solid diluents can be water-soluble or water-insoluble. Water-soluble solid 15 diluents include, but are not limited to, salts such as alkali metal phosphates (e.g., sodium dihydrogen phosphate), alkaline earth phosphates, sulfates of sodium, potassium, magnesium and zinc, sodium and potassium chloride, sodium acetate, sodium carbonate and sodium benzoate, and sugars and sugar derivatives such as sorbitol, lactose, sucrose and mannitol, and mixtures thereof. Examples of water-insoluble solid diluents include, 20 but are not limited to clays, synthetic and diatomaceous silicas, calcium and magnesium silicates, titanium dioxide, aluminum, calcium and zinc oxide, and mixtures of these.

Wetting agents include, but are not limited to, alkyl sulfosuccinates, laureates, alkyl sulfates, phosphate esters, acetylenic diols, ethoxyfluorinated alcohols, ethoxylated silicones, alkyl phenol ethoxylates, benzene sulfonates, alkyl-substituted benzene 25 sulfonates, alkyl a-olefin sulfonates, naphthalene sulfonates, alkyl-substituted naphthalene sulfonates, condensates of naphthalene sulfonates and alkyl-substituted naphthalene sulfonates with formaldehyde, and alcohol ethoxylates, and mixtures of these. Alkyl naphthalene sulphonates, sodium salts are particularly useful for the composition of the invention

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Dispersants include, but are not limited to, sodium, calcium and ammonium salts of ligninsulfonates (optionally polyethoxylated); sodium and ammonium salts of maleic anhydride copolymers; sodium salts of condensed phenolsulfonic acid; and naphthalene sulfonate-formaldehyde condensates, and mixtures of these. Ligninsulfonates such as
5 sodium ligninsulfonates are particularly useful for the composition of the invention. Naphthalene sulfonate-formaldehyde condensates, such as naphthalenesulfonic acid, polymers with formaldehyde, and sodium salts are particularly useful for the composition of an embodiment of the invention

Thickening agents include, but are not limited to, guar gum, pectin, casein,
10 carrageenan, xanthan gum, alginates, methylcellulose, hydroxyethylcellulose, hydroxypropylcellulose, and carboxymethylcellulose, and mixtures of these. Synthetic thickening agents include derivatives of the former categories, and also polyvinyl alcohols, polyacrylamides, polyvinylpyrrolidones, various polyethers, their copolymers as well as polyacrylic acids and their salts, and mixtures of these. Alkylpolyvinylpyrrolidones are
15 particularly useful for the composition of the present invention

Other formulation ingredients can also be used in the present invention such as dyes, defoamers, drying agents, and the like. These ingredients are known to one skilled in the art.

The crystalline modification I of isoxadifen-ethyl according to an embodiment of
20 the invention can be present in formulations and in its use forms, prepared from these formulations, and as a safener to reduce the phototoxicity of sulfonylurea herbicides.

All plants, plant parts, and plant loci can be treated in accordance with the invention. In the present context, plants are to be understood as meaning all plants and plant populations such as desired and undesired wild plants or crop plants (including naturally
25 occurring crop plants). Crop plants can be plants which can be obtained by conventional breeding and optimization methods, by biotechnological and genetic engineering methods, or by combinations of these methods, including the transgenic plants and the plant cultivars which can or cannot be protected by plant breeders' rights. Plant parts are to be understood as meaning all parts and organs of plants above and below the ground, such as

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shoot, leaves, needles, stalks, stems, flowers, fruit bodies, fruits, seeds, roots, tubers and rhizomes. Harvested materials, and vegetative and generative propagation materials, for example, cutting, tubers, meristem tissue, rhizomes, offsets, seeds, single and multiple plant cells and any other plant tissues, are also included. Loci of plants include the
5 environment near the plant and/or plant propagation materials, such as the soil in which the plant grows or will be sown, or the plant's surroundings.

As used herein, the term "about," when used in connection with a numerical amount or range, means somewhat more or somewhat less than the stated numerical amount or range, to a deviation of $\pm 10\%$ of the stated numerical amount or endpoint of the
10 range.

The term "herbicidally effective amount" as used herein, refers to the quantity of such a compound or combination of such compounds that is capable of producing a controlling effect on the growth of plants. The controlling effects include all deviation
15 from the natural development of the target plants, for example killing, retardation of one or more aspects of the development and growth of the plant, leaf burn, albinism, dwarfing and the like. Treatment of the plants and plant parts with the compositions or formulations of the inventions according to the invention is carried out directly or by allowing the compositions or formulations to act on their loci, habitat or storage space by the customary
20 treatment methods. Examples of these customary treatment methods include dipping, spraying, vaporizing, fogging, broadcasting, painting on in the case of propagation material, and applying one or more coats particularly in the case of seed.

The benefits of the invention are seen most when the herbicidal composition is applied to kill weeds in growing crops of useful plants, such as maize (corn) including
25 field corns, pop corns and sweet corns, cotton, wheat, barley, rye, triticale, rice, oats, potatoes, sugar beets, plantation crops (such as bananas, fruit trees, rubber trees, tree nurseries), vines, asparagus, bushberries (such as blueberries), caneberries, cranberries, flax, grain sorghum, okra, peppermint, rhubarb, spearmint and sugarcane. In this invention, treatment of growing crops of maize and potatoes are particularly beneficial.

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All percentages are given in weight % unless otherwise indicated.

Embodiments of the invention will now be described by way of the following examples which are provided for illustrative purposes only, and not intended to limit the scope of the disclosure or of the appended claims.

5

Examples

Example 1: Preparation of amorphous isoxadifen-ethyl in accordance with the disclosure of U.S. Patent No. 5,516,750

13.52 g (0.075 mol) of 1,1-diphenylethene and 5.06 g (0.05 mol) of triethylamine are
10 dissolved in 200 ml of ether at 0 °C, and 7.58 g (0.05 mol) of ethyl
2-chloro-2-hydroximinoacetate, dissolved in 100 ml of ether, are subsequently added
dropwise in the course of approximately two hours. After stirring has been continued for
one hour at room temperature, 100 ml of H₂O are added, and the mixture is subsequently
extracted using ether. After drying over a MgSO₄, the ether is distilled off and the residue
15 is purified over a silica gel column (eluent: n-heptane:ethyl acetate=8:2). In this way, 12.7
g of product of melting point 78 °C to 81 °C are obtained.

As shown in Figure 4, the X-ray powder diffraction pattern of the resulting
isoxadifen-ethyl products prepared above has no significant signals, which indicates the
isoxadifen-ethyl product prepared in accordance with the disclosure of U.S. Patent No.
20 5,516,750 is amorphous.

Example 2: Preparation of the crystalline modification I of isoxadifen-ethyl

Crystallization from methanol

10ml methanol was charged into the reactor to dissolve crude, amorphous
25 isoxadifen-ethyl prepared in Example 1 under stirring. This process lasted for 2 hours
under room temperature, and white solid precipitate appeared. The mixture was then
cooled down to 0 °C - 5 °C and maintained at this temperature for 1 hour to allow
complete crystallization. After that, the mixture was centrifuged. The filter cake was

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washed with methanol. The resulting solid was dried under high vacuum to give crystals of pure isoxadifen-ethyl technical (Purity: 98%).

The crystals were characterized as being of the crystalline modification I of isoxadifen-ethyl using both IR spectrometry, X-ray powder diffraction and DSC.

- 5 The IR spectrum of the crystalline modification I of isoxadifen-ethyl is set out in Figure 1. The IR spectrum exhibits characteristic peaks at 2984.31, 2928.19, 2160.89, 1978.81 and 1716.65 cm^{-1} .

The melting point of the product was measured by DSC and exhibits a peak at 86.5 °C as shown in Figure 3.

- 10 The crystalline modification I of isoxadifen-ethyl has the X-ray powder diffractogram shown in Figure 2 with the reflexes listed in Table 1 below.

Table 1

Crystalline modification I	
2 θ (°)	d (Å)
11.027 $\pm$ 0.2	9.31 $\pm$ 0.05
17.841 $\pm$ 0.2	5.77 $\pm$ 0.05
19.492 $\pm$ 0.2	5.28 $\pm$ 0.05
19.841 $\pm$ 0.2	5.19 $\pm$ 0.05
24.801 $\pm$ 0.2	4.17 $\pm$ 0.05
25.775 $\pm$ 0.2	4.01 $\pm$ 0.05
26.895 $\pm$ 0.2	3.85 $\pm$ 0.05
27.813 $\pm$ 0.2	3.72 $\pm$ 0.05

Example 3: Preparation of the crystalline modification I of isoxadifen-ethyl

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Crystallization from acetonitrile

10ml acetonitrile was charged into the reactor to dissolve crude, amorphous isoxadifen-ethyl prepared in Example 1 under stirring. This process lasted for 2 hours under room temperature, and white solid precipitate appeared. The mixture was then cooled down to 0 °C - 5 °C and maintained at this temperature for 1 hour to allow complete crystallization. After that, the mixture was centrifuged. The filter cake was washed with some acetonitrile. The resulting solid was dried under high vacuum to give crystals of pure isoxadifen-ethyl technical (Purity: 98%).

The crystals were characterized as being the crystalline modification I of isoxadifen-ethyl using IR spectrometry, X-ray powder diffraction and DSC as described in Example 2.

Example 4: Preparation of oil based suspension concentrate (OD) formulation

All the components listed in Table 2 below were mixed uniformly and ground with a Dyno-Mill (manufactured by Willy A. Bachofen AG) to obtain an oil based suspension concentrate.

Table 2

Ingredients	Weights %		Function
Isoxadifen-ethyl, crystalline modification I, 98% (prepared in Example 2)	10.20	0.00	Safener
Amorphous isoxadifen-ethyl (prepared in Example 1)	0.00	10.20	Safener
Tribenuron-methyl, 98%	40.80	40.80	Active compound
Sodium lignosulfonate (REAX [®] 88B)	12.00	12.00	Dispersing agent
Alkylpolyvinylpyrrolidone	10.00	10.00	Thickening agent
Corn oil	Balance to 100%	Balance to 100%	Carrier

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Example 5: Preparation of soluble granules (SG)

5 All the components listed in Table 3 below were mixed, blended and milled in a high-speed rotary mill. Sufficient water was added to obtain an extrudable paste. The paste was extruded through a die or screen to form an extrudate. The wet extrudate was dried at 70°C in a vacuum oven and then sifted through 0.71 mm – 2 mm screens to obtain the product granules.

Table 3

Ingredients	Weights %		Function
Isoxadifen-ethyl, crystalline modification I, 98% (prepared in Example 2)	7.14	0.00	Safener
Amorphous isoxadifen-ethyl (prepared in Example 1)	0.00	7.14	Safener
Metsulfuron-methyl, 98%	25.51	25.51	Active compound
Lignosulfonic acid, sodium salt, (REAX® 88B)	15.00	15.00	Dispersing agent
Naphthalenesulfonic acid, polymer with formaldehyde, sodium salt (TAMOL® NN 8906)	6.00	6.00	Dispersing agent
Sodium acetate	4.00	4.00	Filler
Sodium carbonate	4.00	4.00	Filler
Non-ionic aqueous emulsion of Polydimethylsiloxanes	1.00	1.00	Antifoaming agent
Mannitol	Balance to 100%	Balance to 100%	carrier

Example 6: Preparation of water dispersible granules (WG)

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5 All the components listed in Table 4 below were mixed, blended and milled in a high-speed rotary mill. Sufficient water was added to obtain an extrudable paste. The paste was extruded through a die or screen to form an extrudate. The wet extrudate was dried at 70°C in a vacuum oven and then sifted through 0.71 mm – 2 mm screens to obtain the product granules.

Table 4

Ingredients	Weights %		Function
Isoxadifen-ethyl, crystalline modification I, 98% (prepared in Example 2)	7.14	0.00	Safener
Amorphous isoxadifen-ethyl (prepared in Example 1)	0.00	7.14	Safener
Metsulfuron-methyl, 98%	25.51	25.51	Active compound
Alkyl naphthalene sulphonate, sodium salt (Akzo Nobel)	2	2	Wetting agent
Lignosulfonic acid, sodium salt, REAX [®] 88B)	15	15	Dispersing agent
Naphthalenesulfonic acid, polymer with formaldehyde, sodium salt (TAMOL [®] NN8906)	6	6	Dispersing agent
Sucrose	10	10	Filler
Non-ionic aqueous emulsion of Polydimethylsiloxanes	1	1	Antifoaming agent
Mannitol	Balance to 100%	Balance to 100%	Carrier

10 Example 7: Stability test

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The stability of the isoxadifen-ethyl in these compositions was determined by aging samples in heated ovens having the same atmosphere therein, and then comparing the isoxadifen-ethyl content before and after aging to determine relative percentage of hydrolysis (RPH). RPH was calculated by the following equation:

$$RPH = \frac{(The\ final\ weight\ \% \ of\ isoxadifen - ethyl - The\ initial\ weight\ \% \ of\ isoxadifen - ethyl)}{The\ initial\ weight\ \% \ of\ isoxadifen - ethyl} \times 100\%$$

5

Isoxadifen-ethyl content was determined by assaying the compositions with high-pressure liquid chromatography (HPLC) using reverse phase columns and eluants.

Samples prepared in Examples 4, 5 and 6 were stored at 54 °C for 1 week. The procedures are followed according to CIPAC MT 46.3. The concentration of isoxadifen-ethyl was measured at the end of each storage time by HPLC. The results are listed in Table 5.

Table 5.

Sample	Compound	Weight (%) of Isoxadifen-ethyl	RPH%
Example 4	Isoxadifen-ethyl, crystalline modification I	10	0
	Amorphous isoxadifen-ethyl	10	50
Example 5	Isoxadifen-ethyl, crystalline modification I	7	0
	Amorphous isoxadifen-ethyl	7	40
Example 6	Isoxadifen-ethyl, crystalline modification I	7	0
	Amorphous isoxadifen-ethyl	7	45

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CLAIMS

1. A crystalline modification I of isoxadifen-ethyl, exhibiting at least 3 of the following reflexes, in any combination, in an X-ray powder diffractogram recorded using Cu—K α radiation at 25 °C:

5	$2\theta = 11.027 \pm 0.2$	(1)
	$2\theta = 17.841 \pm 0.2$	(2)
	$2\theta = 19.492 \pm 0.2$	(3)
	$2\theta = 19.841 \pm 0.2$	(4)
	$2\theta = 20.583 \pm 0.2$	(5)
10	$2\theta = 22.151 \pm 0.2$	(6)
	$2\theta = 22.920 \pm 0.2$	(7)
	$2\theta = 23.541 \pm 0.2$	(8)
	$2\theta = 24.801 \pm 0.2$	(9)
	$2\theta = 25.775 \pm 0.2$	(10)
15	$2\theta = 26.895 \pm 0.2$	(11)
	$2\theta = 27.813 \pm 0.2$	(12)
	$2\theta = 29.408 \pm 0.2$	(13)
	$2\theta = 30.065 \pm 0.2$	(14)
	$2\theta = 31.228 \pm 0.2$	(15)
20	$2\theta = 33.502 \pm 0.2$	(16)

2. The crystalline modification I of isoxadifen-ethyl according to claim 1, exhibiting at least 3 of the following reflexes, in any combination, in an X-ray powder diffractogram recorded using Cu—K α radiation at 25 °C:

25	$2\theta = 11.027 \pm 0.2$	(1)
----	----------------------------	-----

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$$2\theta = 17.841 \pm 0.2 \quad (2)$$

$$2\theta = 19.492 \pm 0.2 \quad (3)$$

$$2\theta = 19.841 \pm 0.2 \quad (4)$$

$$2\theta = 24.801 \pm 0.2 \quad (9)$$

5 $2\theta = 25.775 \pm 0.2 \quad (10)$

$$2\theta = 26.895 \pm 0.2 \quad (11)$$

$$2\theta = 27.813 \pm 0.2 \quad (12)$$

3. The crystalline modification I of isoxadifen-ethyl according to claim 1, exhibiting at least four of said reflexes, in any combination.

10 4. The crystalline modification I of isoxadifen-ethyl according to claim 1, exhibiting at least five of said reflexes, in any combination.

5. The crystalline modification I of isoxadifen-ethyl according to claim 1, exhibiting an IR spectrum with one or more of the characteristic bands at 2984.31, 2928.19, 2160.89, 1978.81 and 1716.65 cm^{-1} .

15 6. The crystalline modification I of isoxadifen-ethyl according to claim 1, exhibiting a DSC spectrum having an endothermic peak at 86.5°C .

7. A process for the preparation of a crystalline modification I of isoxadifen-ethyl according to claim 1, comprising:

i) preparing a solution of an amorphous isoxadifen-ethyl in a solvent;

20 ii) effecting crystallization of isoxadifen-ethyl from the solution to obtain a solid precipitate; and

iii) isolating the solid precipitate.

8. The process according to claim 7, wherein the solvent comprises an alcohol, acetonitrile, or a combination thereof.

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9. The process according to Claim 8, wherein the solvent is methanol, acetonitrile, or a mixture thereof.

10. A composition comprising a safening amount of the crystalline modification I of isoxadifen-ethyl according to claim 1, a herbicidally effective amount of at least one
5 sulfonylurea herbicide, and at least one herbicidally acceptable auxiliary.

11. The composition in claim 10, in the form of a suspension concentrate (SC), an oil-based suspension concentrate (OD), a water-soluble granule (SG), a dispersible concentrate (DC), an emulsifiable concentrate (EC), an emulsion seed dressing, a suspension seed dressing, a granule (GR), a microgranule (MG), a suspoemulsion (SE) or
10 a water-dispersible granule (WG).

12. The composition according to claim 11, in the form of an oil-based suspension concentrate (OD).

13. The composition according to claim 11, in the form of a water-soluble granule (SG).

14. The composition according to claim 11, in the form of a water-dispersible granule (WG).
15

15. The composition according to claim 10, wherein the auxiliary is selected from one or more of a solvent, a diluent, a wetting agent, a dispersant, and a thickening agent and an antifoaming agent.

16. The composition according to claim 10, where the weight ratio of the isoxadifen-ethyl to sulfonylurea herbicides is in a range of about 1:10 to about 10:1, in particular in a range of about 1:10 to about 5:1.
20

17. A method of controlling unwanted plant growth, comprising applying to the plant an effective amount of a composition according to claim 10.

18. The composition according to claim 10, wherein the sulfonylurea herbicide comprises one or more selected from the group consisting of amidosulfuron, azimsulfuron, bensulfuron-methyl, chlorimuron-ethyl, chlorsulfuron, cinosulfuron, cyclosulfamuron, ethametsulfuron-methyl, ethoxysulfuron, flupyrsulfuron-methyl, flazasulfuron,
25

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foramsulfuron, halosulfuron-methyl, imazosulfuron, iodosulfuron-methyl,
mesosulfuron-methyl, metsulfuron-methyl, nicosulfuron, oxasulfuron,
primisulfuron-methyl, prosulfuron, pyrazosulfuron-ethyl, rimsulfuron,
sulfometuron-methyl, sulfosulfuron, thifensulfuron-methyl, triasulfuron, tribenuron-methyl,
5 trifloxysulfuron, triflusulfuron-methyl, tritosulfuron and salts thereof, and mixtures
thereof.

19. The composition according to claim 18, wherein the sulfonylurea herbicide
comprises one or more selected from the group consisting of bensulfuron-methyl,
flupyralsulfuron-methyl, metsulfuron-methyl, nicosulfuron, rimsulfuron,
10 sulfometuron-methyl, thifensulfuron-methyl, tribenuron-methyl, halosulfuron-methyl,
iodosulfuron-methyl, mesosulfuron-methyl and salts thereof, and mixtures thereof.

20. Crystalline modification I of isoxadifen-ethyl substantially as hereinbefore
described having reference to either of Figures 1 or 2.

21. A process for preparing modification I of isoxadifen-ethyl substantially as
15 hereinbefore described.

22. A composition comprising modification I of isoxadifen-ethyl substantially as
hereinbefore described.

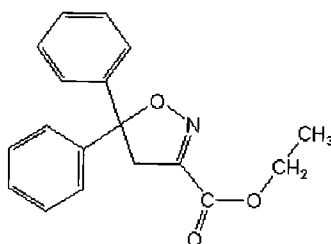
23. A use of modification I of isoxadifen-ethyl substantially as hereinbefore
described.

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ABSTRACT

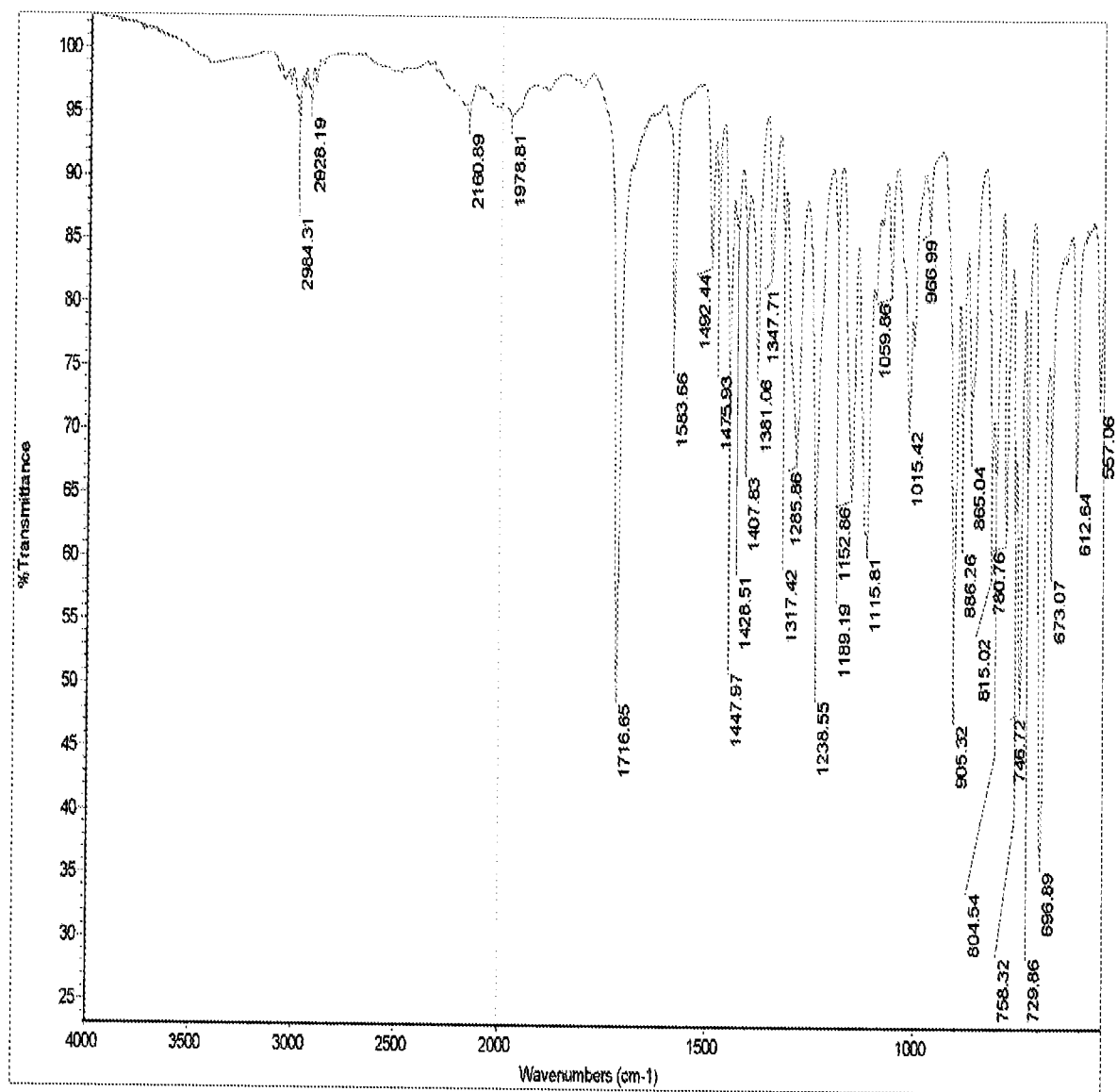
A crystalline form of isoxadifen-ethyl of formula (I), the crystal preparation process, the analyses of the crystal through various analytical methods and using the crystal to prepare stable agrochemical formulation. The invention also describes the use of
5 various solvents towards the crystalline form preparation conditions.



(I)

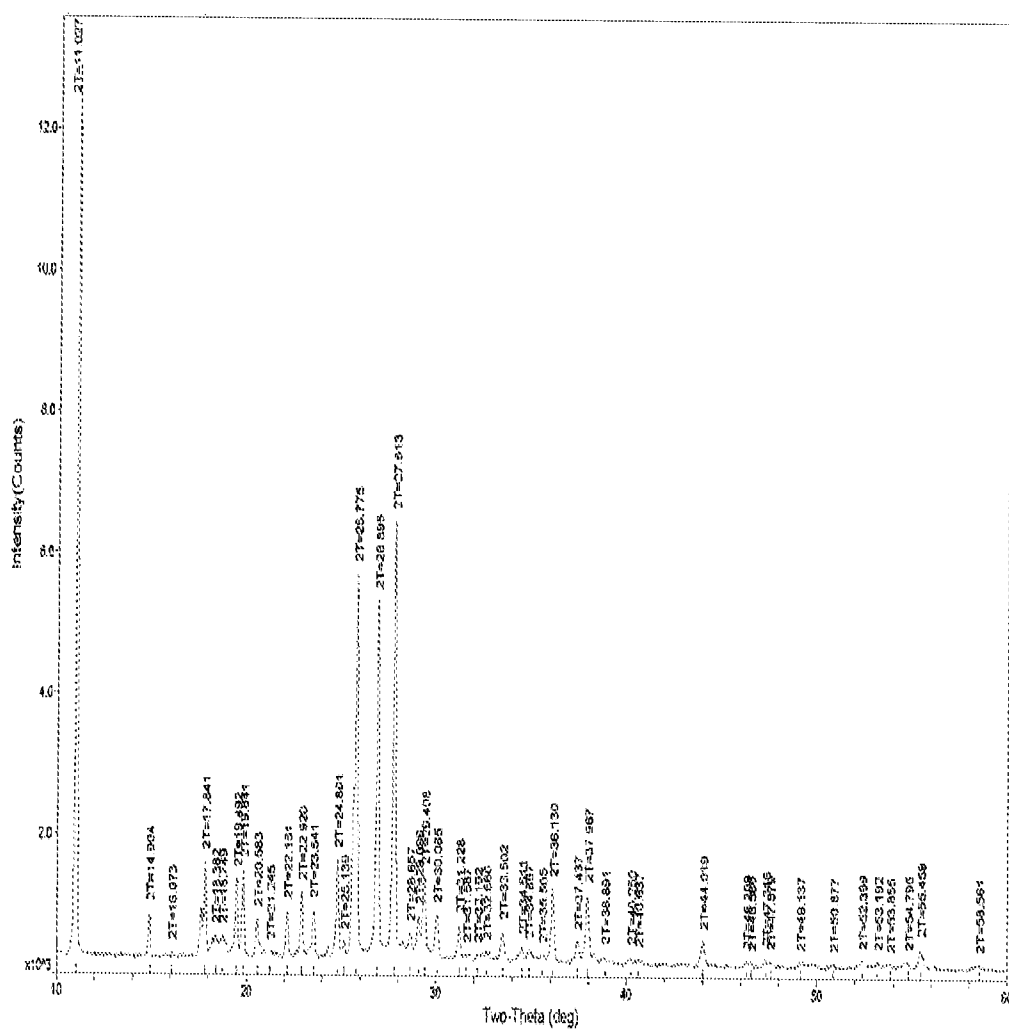
PA-0413

Figure 1



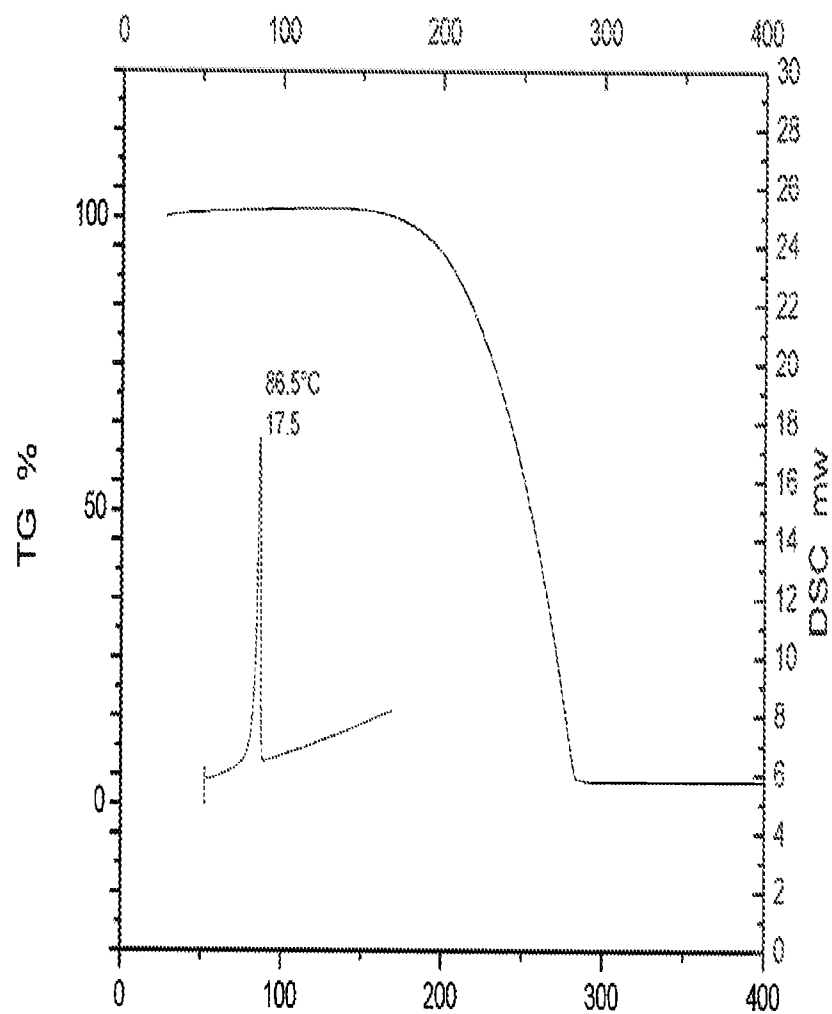
PA-0413

Figure 2



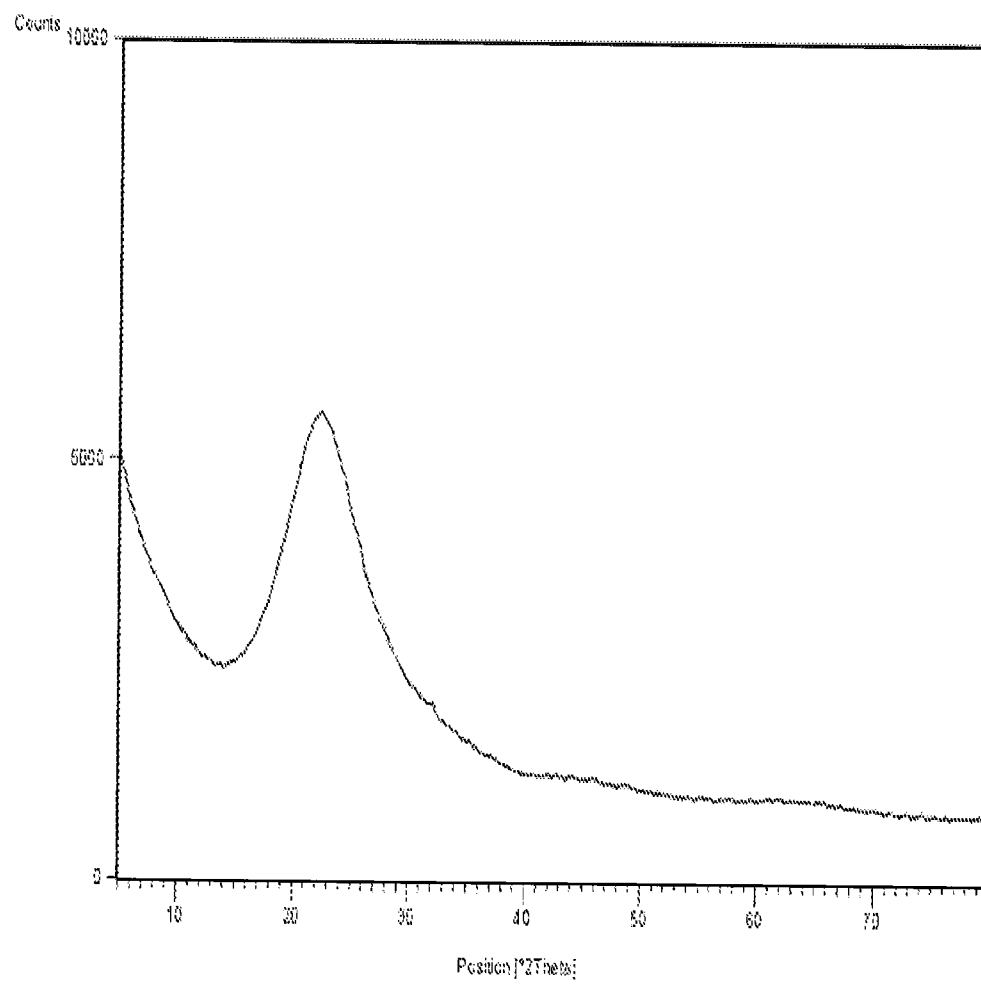
PA-0413

Figure 3



PA-0413

Figure 4



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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	727440-01028
		Application Number	
Title of Invention	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME		
The application data sheet is part of the provisional or nonprovisional application for which it is being submitted. The following form contains the bibliographic data arranged in a format specified by the United States Patent and Trademark Office as outlined in 37 CFR 1.76. This document may be completed electronically and submitted to the Office in electronic format using the Electronic Filing System (EFS) or the document may be printed and included in a paper filed application.			

Secrecy Order 37 CFR 5.2

☐ Portions or all of the application associated with this Application Data Sheet may fall under a Secrecy Order pursuant to 37 CFR 5.2 (Paper filers only. Applications that fall under Secrecy Order may not be filed electronically.)

Inventor Information:

Inventor 1					Remove
Legal Name					
Prefix	Given Name	Middle Name	Family Name	Suffix	
	James	Timothy	BRISTOW		
Residence Information (Select One) ☐ US Residency ☒ Non US Residency ☐ Active US Military Service					
City	Chai Wan, Hong Kong		Country of Residence i	CN	
Mailing Address of Inventor:					
Address 1		Unit 6, 26/F, E-Trade Plaza, 29 Lee Chung Street			
Address 2					
City	Chai Wan, Hong Kong		State/Province		
Postal Code			Country i	CN	
All Inventors Must Be Listed - Additional Inventor Information blocks may be generated within this form by selecting the Add button.					

Correspondence Information:

Enter either Customer Number or complete the Correspondence Information section below. For further information see 37 CFR 1.33(a).			
☐ An Address is being provided for the correspondence Information of this application.			
Customer Number	35161		
Email Address			Add Email Remove Email

Application Information:

Title of the Invention	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME		
Attorney Docket Number	727440-01028	Small Entity Status Claimed	☐
Application Type	Nonprovisional		
Subject Matter	Utility		
Total Number of Drawing Sheets (if any)	4	Suggested Figure for Publication (if any)	

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	727440-01028
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Title of Invention	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME		

Filing By Reference :

Only complete this section when filing an application by reference under 35 U.S.C. 111(c) and 37 CFR 1.57(a). Do not complete this section if application papers including a specification and any drawings are being filed. Any domestic benefit or foreign priority information must be provided in the appropriate section(s) below (i.e., "Domestic Benefit/National Stage Information" and "Foreign Priority Information").

For the purposes of a filing date under 37 CFR 1.53(b), the description and any drawings of the present application are replaced by this reference to the previously filed application, subject to conditions and requirements of 37 CFR 1.57(a).

Application number of the previously filed application	Filing date (YYYY-MM-DD)	Intellectual Property Authority or Country

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☐ Request Early Publication (Fee required at time of Request 37 CFR 1.219)

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Representative information should be provided for all practitioners having a power of attorney in the application. Providing this information in the Application Data Sheet does not constitute a power of attorney in the application (see 37 CFR 1.32). Either enter Customer Number or complete the Representative Name section below. If both sections are completed the customer Number will be used for the Representative Information during processing.

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Customer Number	35161		

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This section allows for the applicant to either claim benefit under 35 U.S.C. 119(e), 120, 121, or 365(c) or indicate National Stage entry from a PCT application. Providing this information in the application data sheet constitutes the specific reference required by 35 U.S.C. 119(e) or 120, and 37 CFR 1.78.

When referring to the current application, please leave the application number blank.

Prior Application Status		Remove	
Application Number	Continuity Type	Prior Application Number	Filing Date (YYYY-MM-DD)

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	727440-01028
		Application Number	
Title of Invention	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME		

This section allows for the applicant to claim priority to a foreign application. Providing this information in the application data sheet constitutes the claim for priority as required by 35 U.S.C. 119(b) and 37 CFR 1.55(d). When priority is claimed to a foreign application that is eligible for retrieval under the priority document exchange program (PDX) the information will be used by the Office to automatically attempt retrieval pursuant to 37 CFR 1.55(h)(1) and (2). Under the PDX program, applicant bears the ultimate responsibility for ensuring that a copy of the foreign application is received by the Office from the participating foreign intellectual property office, or a certified copy of the foreign priority application is filed, within the time period specified in 37 CFR 1.55(g)(1).

			Remove
Application Number	Country ⁱ	Filing Date (YYYY-MM-DD)	Access Code ^j (if applicable)
Additional Foreign Priority Data may be generated within this form by selecting the Add button.			Add

Statement under 37 CFR 1.55 or 1.78 for AIA (First Inventor to File) Transition Applications

☐ This application (1) claims priority to or the benefit of an application filed before March 16, 2013 and (2) also contains, or contained at any time, a claim to a claimed invention that has an effective filing date on or after March 16, 2013. NOTE: By providing this statement under 37 CFR 1.55 or 1.78, this application, with a filing date on or after March 16, 2013, will be examined under the first inventor to file provisions of the AIA.

Authorization to Permit Access:

☐ Authorization to Permit Access to the Instant Application by the Participating Offices

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	727440-01028
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If checked, the undersigned hereby grants the USPTO authority to provide the European Patent Office (EPO), the Japan Patent Office (JPO), the Korean Intellectual Property Office (KIPO), the World Intellectual Property Office (WIPO), and any other intellectual property offices in which a foreign application claiming priority to the instant patent application is filed access to the instant patent application. See 37 CFR 1.14(c) and (h). This box should not be checked if the applicant does not wish the EPO, JPO, KIPO, WIPO, or other intellectual property office in which a foreign application claiming priority to the instant patent application is filed to have access to the instant patent application.

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Clear			
☒ Assignee	☐ Legal Representative under 35 U.S.C. 117	☐ Joint Inventor	
☐ Person to whom the inventor is obligated to assign.		☐ Person who shows sufficient proprietary interest	
If applicant is the legal representative, indicate the authority to file the patent application, the inventor is:			
Name of the Deceased or Legally Incapacitated Inventor :			
If the Applicant is an Organization check here. ☒			
Organization Name	ROTAM AGROCHEM INTERNATIONAL CO., LTD		
Mailing Address Information:			
Address 1	Unit 6, 26/F, E-Trade Plaza, 29 Lee Chung Street		
Address 2	Chai Wan, Hong Kong		
City		State/Province	
Country ⁱ	CN	Postal Code	
Phone Number		Fax Number	

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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	727440-01028
		Application Number	
Title of Invention	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME		
Email Address			
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Organization Name	ROTAM AGROCHEM INTERNATIONAL CO., LTD		
Mailing Address Information For Assignee including Non-Applicant Assignee:			
Address 1	Unit 6, 26/F, E-Trade Plaza, 29 Lee Chung Street		
Address 2			
City	Chai Wan, Hong Kong	State/Province	
Country i	CN	Postal Code	
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Email Address			
Additional Assignee or Non-Applicant Assignee Data may be generated within this form by selecting the Add button. Add			

Signature:

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Signature	/Bruce D. Gray/		Date (YYYY-MM-DD)	2015-10-29	
First Name	Bruce	Last Name	Gray	Registration Number	35799
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Application Data Sheet 37 CFR 1.76		Attorney Docket Number	727440-01028
		Application Number	
Title of Invention	A NOVEL FORM OF ISOXADIFEN-ETHYL, A PROCESS FOR ITS PREPARATION AND USE OF THE SAME		

This collection of information is required by 37 CFR 1.76. The information is required to obtain or retain a benefit by the public which is to file (and by the USPTO to process) an application. Confidentiality is governed by 35 U.S.C. 122 and 37 CFR 1.14. This collection is estimated to take 23 minutes to complete, including gathering, preparing, and submitting the completed application data sheet form to the USPTO. Time will vary depending upon the individual case. Any comments on the amount of time you require to complete this form and/or suggestions for reducing this burden, should be sent to the Chief Information Officer, U.S. Patent and Trademark Office, U.S. Department of Commerce, P.O. Box 1450, Alexandria, VA 22313-1450. DO NOT SEND FEES OR COMPLETED FORMS TO THIS ADDRESS. **SEND TO: Commissioner for Patents, P.O. Box 1450, Alexandria, VA 22313-1450.**

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7. A record from this system of records may be disclosed, as a routine use, to the Administrator, General Services, or his/her designee, during an inspection of records conducted by GSA as part of that agency's responsibility to recommend improvements in records management practices and programs, under authority of 44 U.S.C. 2904 and 2906. Such disclosure shall be made in accordance with the GSA regulations governing inspection of records for this purpose, and any other relevant (i.e., GSA or Commerce) directive. Such disclosure shall not be used to make determinations about individuals.
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